IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

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| Applicant | : | Rolf Wiedermann et al. |
| Serial No. | : | 08/362,547 |
| Filed | : | January 3, 1995 |
| For | : | A process for the production of rigid foams containing urethane groups and predominantly isocyanurate groups |
| Art Unit | : | 1711 |
| Examiner | : | J. Cooney |

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# DECLARATION

I, Wolfgang Friederichs, a German citizen, residing at Am Ackerrain 16, 50933 Cologne, Germany, declare as follows:

1) that I have studied chemistry at the University of Cologne and received the degree of doctor rer. nat. at this University in 1985;

2) that since 1985 I have been in the employ of Bayer AG of Leverkusen, Germany, as a research chemist; and

3) that in the course of my employment I have gained considerable expertise in the field of polyurethanes; and

4) that I have read, and I am familiar with, the Offic Action dated May 7, 2002, which issued in the patent application bearing U.S. Serial Number 08/362,547, filed January 3, 1995, claiming foreign priority of German Patent Application P 42 22 519.1, which was filed July 9, 1992; and

5) that the following experiments were conducted under my immediate supervision and control:

Comparison Experiment 1. Working example 1 of U.S. Patent Number 5,096,033 ('Volkert') was repeated using as

A Component a mixture comprising:

82.4 parts by weight of a sucrose-based polyoxypropylene polyether polyol having a hydroxyl number of 380,

3.6 parts by weight of water,

2.3 parts by weight of N,N-dimethylcyclohexylamine,

0.8 parts by weight of a foam stabilizer based on a silicone (Tegostab® B8421 from Goldschmidt AG, Essen, FRG), and

10.9 parts by weight of cyclopentane;

and as

B component a mixture of diphenylmethane diisocyanates and polyphenyl polymethylene polyisocyanate (polymeric MDI, NCO content 31 weight percent.

100 parts by weight of the A component and 148 parts by weight of the B component (corresponding to a NCO/OH index of 114) were thoroughly mixed at 23°C using a high speed stirrer at 2000 rpm, then the reaction mixture was poured into an open 20cm x 20 cm x 20 cm paper box and was allowed to foam.

A rigid polyurethane foam having a calculated density of 18 g/cm$^3$ was obtained. This calculated density corresponds to an actual density of 22 g/cm$^3$. The foam was dimensionally stable but had a brittle surface.

Comparison Experiment 2. Comparison Experiment 1 was repeated using a modified A Component which contained no water. The amount of B component was adjusted to establish the same NCO/OH index value as in Experiment 1. In order to obtain a foam of similar density, the proportion of cyclopentane in the A Component had to be increased.

A Component :

82.4 parts by weight of a sucrose-based polyoxypropylene polyether polyol having a hydroxyl number of 380,

2.3 parts by weight of N,N-dimethylcyclohexylamine,

0.8 parts by weight of a foam stabilizer based on a silicone (Tegostab® B8421 from Goldschmidt AG, Essen, FRG), and

24.3 parts by weight of cyclopentane;

100 parts by weight of the A component and 86.3 parts by weight of the B component (corresponding to a NCO/OH index of 114) were thoroughly mixed at 23°C using a high speed stirrer at 2000 rpm, then the reaction mixture was poured into an open 20cm x 20 cm x 20 cm paper box and was allowed to foam.

A rigid polyurethane foam having the same calculated density as the foam of example 1 was obtained. The foam was not dimensionally stable; strong shrinkage was observed.

Comparison Experiment 3. Comparison Experiment 1 was repeated using a modified A Component which contained no water. Additionally, the amount of B component was adjusted to establish a NCO/OH index value of 300. A trimerization catalyst (Desmorapid® 1792, Bayer AG) was added to the A Component, in order to obtain a foam containing

predominantly isocyanurate groups. In order to obtain a foam of similar density, the proportion of cyclopentane in the A Compon nt had to be increased.

A Component :

82.4 parts by weight of a sucrose-based polyoxypropylene polyether polyol having a hydroxyl number of 380,

2.3 parts by weight of N,N-dimethylcyclohexylamine,

5.5 parts by weight of a trimerization catalyst (Desmorapid® 1792)

0.8 parts by weight of a foam stabilizer based on a silicone (Tegostab® B8421 from Goldschmidt AG, Essen, FRG), and

49.9 parts by weight of cyclopentane;

100 parts by weight of the A component and 258.6 parts by weight of the B component (corresponding to a NCO/OH index of 300) were thoroughly mixed at 23°C using a high speed stirrer at 2000 rpm, then the reaction mixture was poured into an open 20cm x 20 cm x 20 cm paper box and was allowed to foam.

A rigid polyurethane foam having the same calculated density as the foam of example 1 was obtained. The foam was dimensionally stable but brittle.

Comparison Experiment 4. Comparison Experiment 1 was repeated using a modified A Component which contained no water. Additionally, the amount of B component was adjusted to establish a NCO/OH index value of 500. A trimerization catalyst (Desmorapid® 1792, Bayer AG) was added to the A Component, in order to obtain a foam containing predominantly isocyanurate groups. In order to obtain a foam of similar density, the proportion of cyclopentane in the A Component had to be increased.

A Component :

82.4 parts by weight of a sucrose-based polyoxypropylen polyether polyol having a hydroxyl number of 380,

2.3 parts by weight of N,N-dimethylcyclohexylamine,

3.0 parts by weight of a trimerization catalyst (Desmorapid® 1792)

0.8 parts by weight of a foam stabilizer based on a silicone (Tegostab® B8421 from Goldschmidt AG, Essen, FRG), and

72.9 parts by weight of cyclopentane;

100 parts by weight of the A component and 407.2 parts by weight of the B component (corresponding to a NCO/OH index of 500) were thoroughly mixed at 23°C using a high speed stirrer at 2000 rpm, then the reaction mixture was poured into an open 20cm x 20 cm x 20 cm paper box and was allowed to foam.

A rigid polyurethane foam having the same calculated density as the foam of example 1 was obtained. The foam was dimensionally stable but very brittle.

6) Taken together with the experimental results presented in the working examples and the comparison examples presented in U.S. patent application Serial Number 08/362,547, these comparison experiments demonstrate that only by using a formulation as claimed in U.S. patent application Serial Number 08/362,547, a hydrocarbon-blown rigid foam containing urethane groups and predominantly isocyanurate groups is obtained which shows no surface brittleness and is dimensionally stable.

The omission of water from the formulation disclosed in the Volkert reference, alone or in combination with increasing the NCO/OH index to the range defined by the claims of U.S. patent application Serial Number 08/362,547, does not lead to foams having satisfactory properties.

I further declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the pending Application Serial Number 08/362,547 or any patent issuing thereon.

Wolfgang Friedrichs

WOLFGANG FRIEDERICHS

Signed at Dormagen, this 24 day of July, 2002